

## 九州工業大学学術機関リポジトリ



Title	Analysis of Magnetization Reversal Process of Nd-Fe-B Sintered Magnets by Magnetic Domain Observation Using Kerr Microscope
Author(s)	Takezawa, M.; Kimura, Y.; Morimoto, Y.; Yamasaki, J.
Issue Date	2013-07-15
URL	<a href="http://hdl.handle.net/10228/5875">http://hdl.handle.net/10228/5875</a>
Rights	IEEE

# Analysis of Magnetization Reversal Process of Nd–Fe–B Sintered Magnets by Magnetic Domain Observation Using Kerr Microscope

Masaaki Takezawa, Yuya Kimura, Yuji Morimoto, and Jiro Yamasaki

Dept. of Applied Science for Integrated System Eng., Faculty of Eng., Kyusyu Institute of Technol., Kitakyushu 804-8550, Japan

We used a Kerr microscope, image processing, and photo editing to clarify magnetization reversal and its propagation in a sintered Nd–Fe–B magnet. Magnetic domain change was observed when a DC field from +20 to –20 kOe was applied to a sintered Nd–Fe–B magnet. Simultaneous magnetization reversal in several grains along the easy axis direction and its propagation to neighboring grains occurred. This indicates that the nucleation field in a grain and magnetic interaction between grains are important controlling factors of the coercivity of sintered Nd–Fe–B magnets.

**Index Terms**—Kerr microscope, sintered Nd–Fe–B magnet, domain structure, image processing

## I. INTRODUCTION

ND–Fe–B permanent magnets are key materials for producing high-power motors used in electrical vehicles. Although thermal stability up to 200 °C is required for electrical vehicles, the coercivity of Nd–Fe–B magnet decreases dramatically at 200 °C. Therefore, a very high coercivity of 30 kOe is necessary at room temperature. To enhance coercivity, commercially available Nd–Fe–B magnets, containing large amounts of the rare earth metal Dy, are used [1], [2]. However, the low natural abundance of Dy is a problem. A Kerr microscope is useful for *in-situ* domain observation in high magnetic fields [3], [4]. In this study, we used a Kerr microscope, image processing, and photo editing to clarify domain reversal propagation. We aim to establish the coercivity mechanism of sintered Nd–Fe–B magnets by magnetic domain observation for realizing Dy-free or Dy-lean Nd–Fe–B magnets with high coercivity.

## II. EXPERIMENTAL

The magnetic domains of commercially available sintered Nd–Fe–B magnet, including approximately 1% Dy, used in a voice coil motor were observed using a Kerr microscope built in an electromagnet that produced a high magnetic field of up to 20 kOe [5]. The magnet was cut into 5-mm-thick, 9-mm-long, and 8-mm-wide samples, and the sample surfaces were polished to clearly reveal domain configuration. In addition, an SiO thin film was deposited on the surfaces as an antireflection coating by vacuum evaporation. The in-plane easy axis was along the width of the sample. The magnets were cut at an angle of 10° with respect to the c-axis for revealing the domain configuration underneath the surface, as described in [6]. The grain size of the magnets ranged from 10 to 30 μm. The demagnetization of a previously magnetized magnet with a pulse field of +50 kOe was observed by applying a DC field from +20 to –20 kOe along the width of the magnet.

For detailed observations of magnetization reversal, we extracted the domain change by subtracting the domain image

in an applied field from that in another applied field using photo editing. Figure 1 shows a schematic view of the image-processing method. Figures 1(a) and (b) show the respective domain images when DC fields of +1.7 and +2.0 kOe were applied. In these figures, the bright and dark domains are magnetized in the rightward and leftward directions, respectively. Dark and bright domains can be observed in a grain at the center of the photo in Fig. 1(a). In Fig. 1(b), the area of the bright domain changed because of domain wall displacement, as can be observed by comparing the area within the ellipse indicated by the dashed line in Figs. 1(a) and (b). Figure 1(c) shows the image-processed photograph after subtracting the domain image of Fig. 1(a) from that of Fig. 1(b). Thus, the area of magnetization reversal due to wall displacement can be determined.

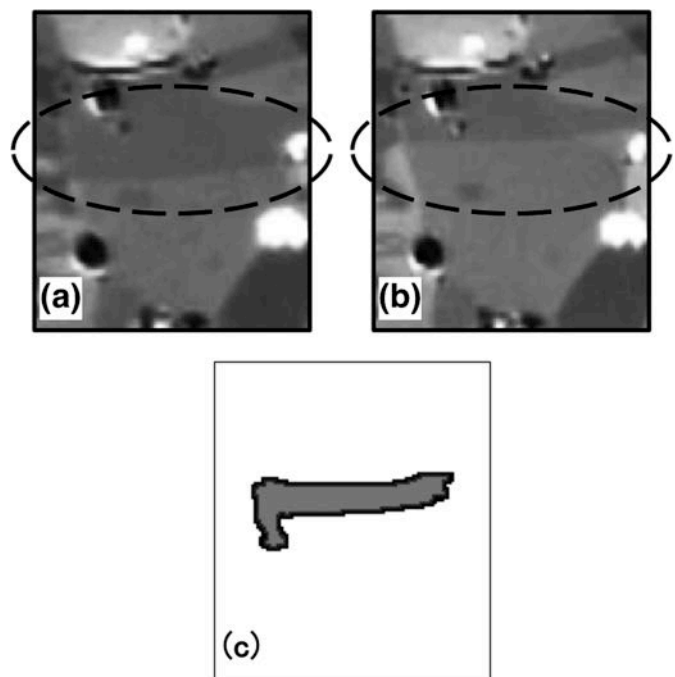


Fig. 1. Image-processing method: (a) domain image of a sintered Nd–Fe–B magnet at a DC field of +1.7 kOe, (b) domain image at a DC field of +2.0 kOe, and (c) image-processed area.

### III. RESULTS AND DISCUSSION

Figure 2 shows the domain images of the sintered Nd-Fe-B magnet when a DC field from +20 to -20 kOe was applied. The bright and dark domains are magnetized in the rightward and leftward directions, respectively. When the DC field decreased from +20 to +5.0 kOe, all grains exhibited bright domains having rightward magnetization components, as shown in Fig. 2(a). The data indicate that a saturated state was obtained in that field. When the DC field decreased to +3.3 kOe, reversed domains were nucleated, and the domain

structure changed from a single domain to a multidomain state within a grain, as indicated by the arrow in Fig. 2(b). The reversed domain grew; furthermore, other domains nucleated and also grew in other grains when the DC field decreased to +1.0 kOe, indicated by the ellipse in Fig. 2(c). Magnetization reversal simultaneously occurred in several grains beyond the grain boundaries along the easy axis direction. Simultaneous magnetization reversal in several grains was also observed when the DC field decreased to zero, marked by the ellipses in Figs. 2(d) and (e). When the DC field reached -2.7 kOe, all

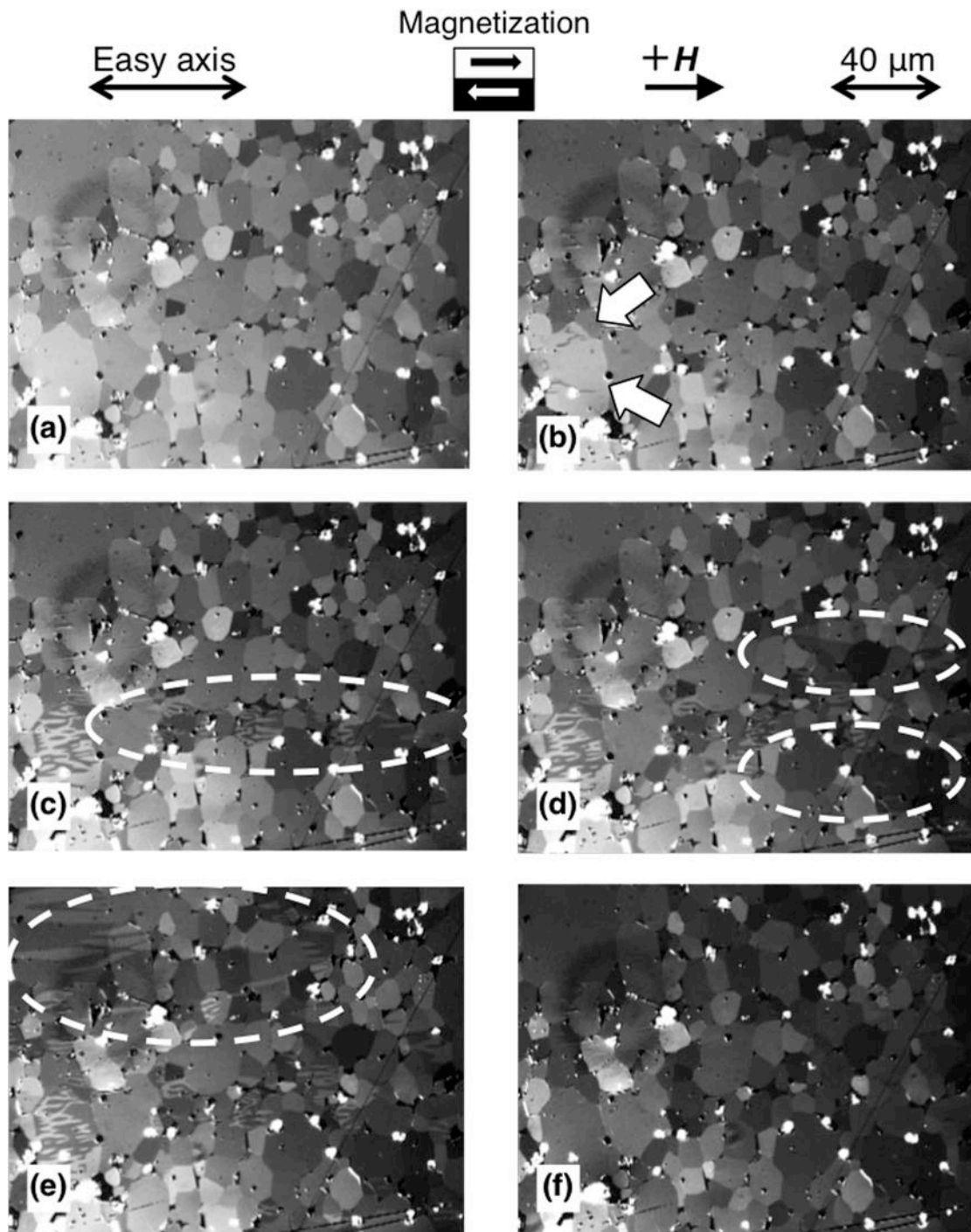


Fig. 2. Domain images of the sintered Nd-Fe-B magnet in a DC field of: (a) +5.0 kOe, (b) +3.3 kOe, (c) +1.0 kOe, (d) +0.6 kOe, (e) +0.2 kOe, and (f) -2.7 kOe.

grains, exhibiting dark domains, were inversely saturated, as shown in Fig. 2(f).

Figure 3(a) shows the image-processed photographs indicating the area of magnetization reversal for 0.5-kOe increments of magnetic field strength in a DC field from +3.5 to 0 kOe during demagnetization. Figures 3(b) and (c) show the image-processed photographs for 0.1-kOe increments of field strength in a DC field from +2.5 to +1.5 kOe and +1.2 to +0.2 kOe, respectively. First, the reversal causes the formation of dark areas, and then, it propagates toward the bright areas. The wall displacement in the grains can be identified in the

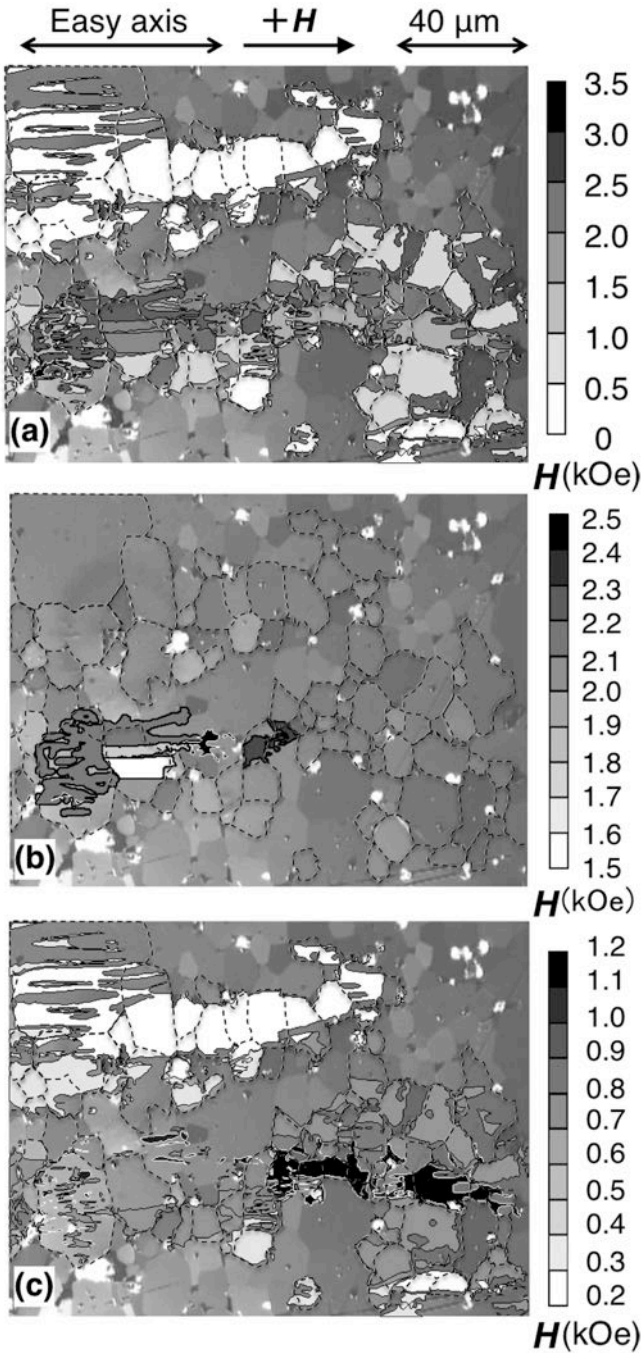


Fig. 3. Image-processed photographs indicating the area of magnetization reversal for magnetic field strength in a DC field from: (a) +3.5 to 0 kOe, (b) +2.5 to +1.5 kOe, and (c) +1.2 to +0.2 kOe.

DC field from +2.2 to +1.5 kOe by image processing, as shown in Fig. 3(b). In Fig. 3(c), dark areas in several grains due to magnetization reversal at +1.2 kOe were observed, and the drastic magnetization reversal propagated above and below several grains at +0.6 kOe. The simultaneous magnetization reversal in several grains along the easy axis direction and its propagation to neighboring grains toward the hard axis direction also occurred in a DC field from +0.3 to +0.2 kOe, indicated by the bright areas in Fig. 3(c). It is difficult to clearly generate magnetization reversal and propagation only by domain imaging with the Kerr microscope, as shown in Fig.

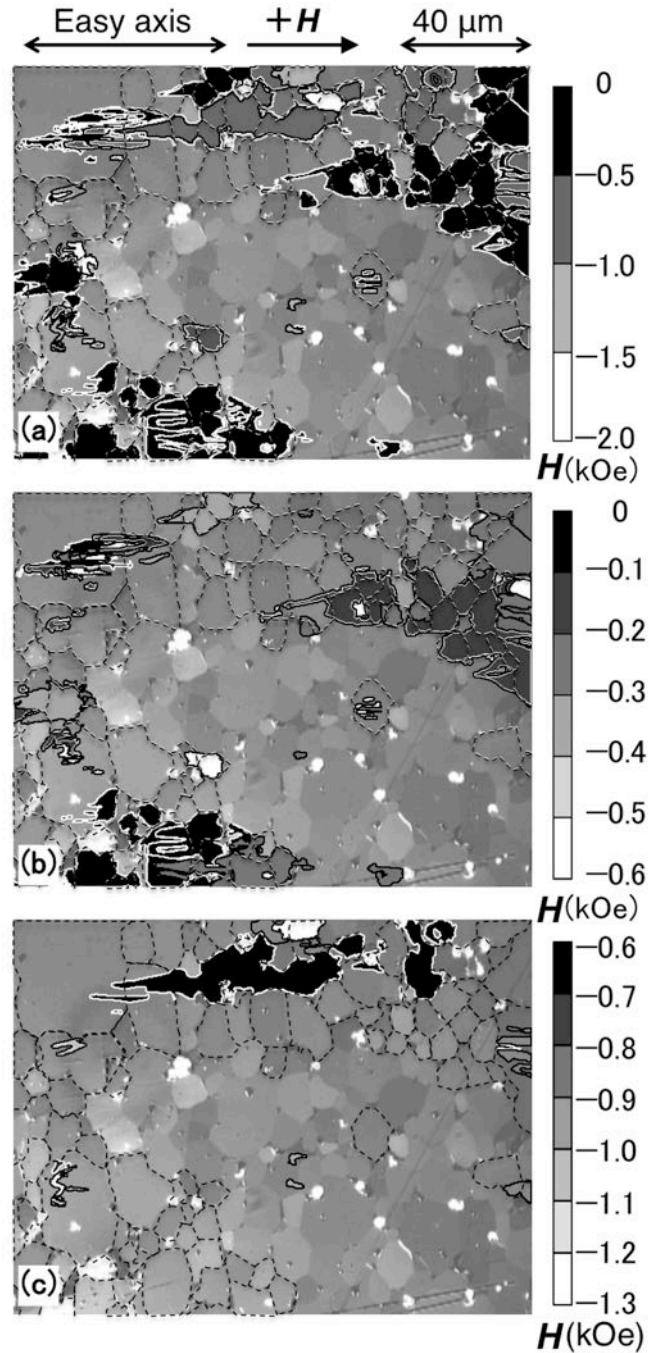


Fig. 4. Image-processed photographs indicating the area of magnetization reversal for magnetic field strength in a DC field from: (a) 0 to -2.0 kOe, (b) 0 to -0.6 kOe, and (c) -0.6 to -1.3 kOe.

2. In contrast, with this image-processing technique, we obtained clear images of magnetization reversal and propagation.

Figure 4(a) shows the image-processed photograph of the area of magnetization reversal for 0.5-kOe increments of magnetic field strength in a DC field from 0 to  $-2.0$  kOe during demagnetization. Figures 4(b) and (c) show the image-processed photographs for 0.1-kOe increments of field strength in a DC field from 0 to  $-0.6$  kOe and  $-0.6$  to  $-1.3$  kOe, respectively. Drastic magnetization reversal along the easy axis direction and its propagation toward neighboring grains beyond grain boundaries were also observed by applying the inverse DC field.

Figure 5(a) shows a wide-field image-processed photograph in a DC field from  $+4.0$  to  $0$  kOe. There are a few hundred grains in the observation area of  $400\text{ }\mu\text{m} \times 300\text{ }\mu\text{m}$  size, as shown in Fig. 5(b). The data show that simultaneous magnetization reversal in several grains and its propagation to neighboring grains also occurred in a wide area of the magnet; this was not a local phenomenon. Because the simultaneous reversal was along the easy axis direction, the magnetization reversal of the sintered Nd-Fe-B magnet occurred beyond the grain boundaries by magnetostatic interaction between grains. Moreover, the propagation of magnetization reversal to neighboring grains along the hard axis direction indicates that the reversal propagated by the exchange interaction between grains and the pinning of domain walls at grain boundaries was insufficient. One of the reasons for the insufficient pinning force at grain boundaries is the removal of the Nd-rich phase by polishing [7]. Therefore, the nucleation field in a grain and the magnetic interaction between grains control the coercivity of sintered Nd-Fe-B magnets.

#### IV. CONCLUSION

In the present study, magnetization reversal and its propagation in sintered Nd-Fe-B magnets were clearly observed by using a Kerr microscope, image processing, and photo editing. We found that simultaneous magnetization reversal in several grains along the easy axis direction and its propagation to neighboring grains occurred. This indicates that the nucleation field in a grain and the magnetic interaction between grains are important in controlling the coercivity of sintered Nd-Fe-B magnets. In the future, image processing will facilitate the investigations of sintered Dy-free or Dy-lean Nd-Fe-B magnets at high temperatures.

#### REFERENCES

- [1] S. Hirosawa, T. Nishiuchi, N. Nozawa, T. Ohkubo, K. Hono, H. Sepehri-Amin, M. Takezawa, J. Yamasaki, S. Yamanuro, T. Tanaka, M. Okano and K. Sumiyama: "Recent Efforts Toward Rare-Metal-Free Permanent Magnets in Japan," *Proceedings of the 21st Workshop on Rare-Earth Permanent Magnets and their Applications*, Bled, Slovenia, pp. 187–192, 2010.
- [2] S. Sugimoto: "Recent Trend of the Researches for Reducing Dysprosium Usage in a Nd-Fe-B Sintered Magnet," *J. Jpn. Soc. Powder and Powder Metallurgy*, Vol.57, pp. 395–400, 2010.
- [3] D. Li and K.J. Strnat: "Domain Behavior in Sintered Nd-Fe-B Magnets during Field-Induced and Thermal Magnetization Change," *J. Appl. Phys.*, Vol.57, pp. 4143–4145, 1985.

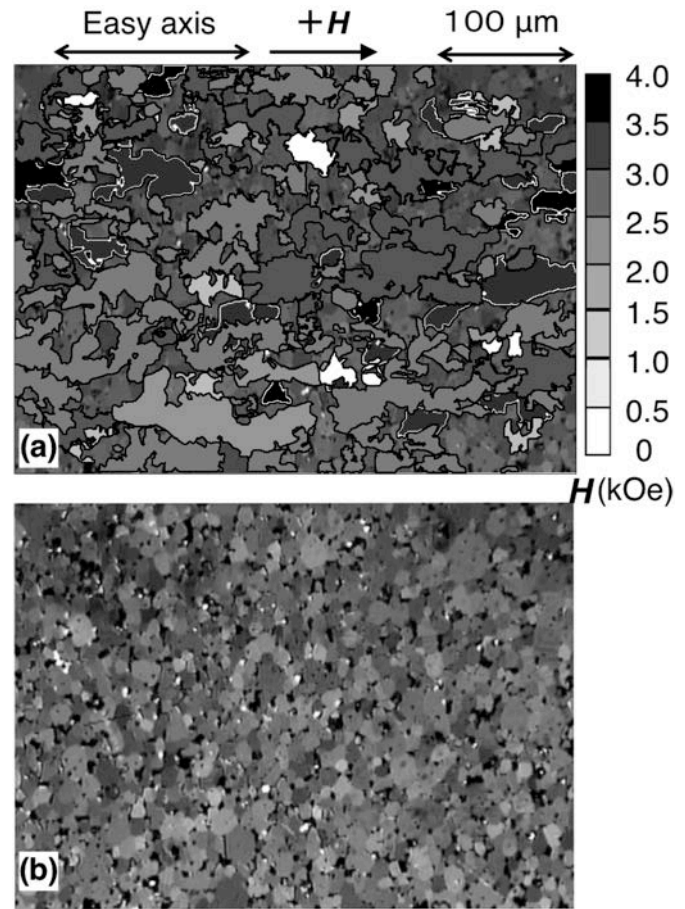


Fig. 5. Area of magnetization reversal for magnetic field strength in a DC field from  $+4.0$  to  $0$  kOe: (a) image-processed and (b) live images.

- [4] M. Takezawa, T. Shimada, S. Kondo, S. Mimura, Y. Morimoto, T. Hidaka and J. Yamasaki: "Domain Observation Technique for Nd-Fe-B Magnet in High Magnetic Field by Image Processing Using Liquid Crystal Modulator," *J. Appl. Phys.*, Vol.101, pp. 09K106, 2007.
- [5] M. Takezawa, N. Tani, Y. Nagashima, Y. Morimoto, J. Yamasaki, N. Nozawa, T. Nishiuchi and S. Hirosawa: "Magnetic Domain Observation of Nd-Fe-B Magnets with Submicron-Sized Grains by High-Resolution Kerr Microscopy," *J. Appl. Phys.*, Vol. 109, pp. 07A709, 2011.
- [6] M. Takezawa, Y. Ichihara, Y. Morimoto and J. Yamasaki: "Surface Domain Configuration of Nd-Fe-B Sintered Magnets Influenced by Underneath Magnetization," *IEEE Trans. Magn.*, Vol.45, pp. 4439–4442, 2009.
- [7] T. Fukagawa and S. Hirosawa: "Coercivity Generation of Surface  $\text{Nd}_2\text{Fe}_{14}\text{B}$  Grains and Mechanism of fcc-Phase Formation at the Nd/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  Interface in Nd-Sputtered Nd-Fe-B Sintered Magnets," *J. Appl. Phys.*, Vol.104, pp. 013911, 2008.